



TITLE:

# The Quantitative Determination of Vanadium in Sea-Water

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characteristics.

The values of  $\epsilon$  and  $\tan \delta$  are also very much sensitive to the temperature. The temperature characteristics shows that both  $\epsilon$  and  $\tan \delta$  become larger with the rise of temperature.

## 7. The Quantitative Determination of Arsenic in Sea-Water

*Masayoshi Ishibashi, Tsunenobu Shigematsu, Yasuharu Nakagawa  
and Yoshihiro Ishibashi*

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The authors have quantitatively determined arsenic in sea-water by Gutzeit method by the following procedure; 1~2 L of sea-water was taken, added 20 mg of  $\text{Fe}^{+++}$  as  $\text{FeCl}_3$  solution, then neutralized with  $\text{NH}_4\text{OH}$  and allowed to stand for 2~3 days. Arsenic in the sample was absorbed by  $\text{Fe}(\text{OH})_3$  and its coprecipitation is nearly complete. The precipitate was filtered and dissolved in 30 ml of  $\text{H}_2\text{SO}_4$  (1:9). This solution was transferred to As determination apparatus, and then arsenic was quantified by Gutzeit method. The results are shown in the following table.

Sea-water		As content $\gamma/\text{L}$ (mean value)	
32.5°N, 135°E	Cl=19.10% (Aug. 20 th. 1949)	3.3	
33°42'40"N, 135°20'E	Cl=19.25% (Feb. 21 th. 1950)	6.0	
35°44'46"N, 135°30'E	Cl=19.12% (Apr. 27 th. 1950)	4.0	
33°42'38"N, 135°19'54"E	Cl=18.77% (Oct. 17 th. 1950)	3.0	

From the above results, As content is found to be 3~6  $\gamma/\text{L}$ , and the mean value 4  $\gamma/\text{L}$ .

The authors are indebted to Dr. T. Tokioka for the help of sampling of sea-water.

## 8. The Quantitative Determination of Vanadium in Sea-Water

*Masayoshi Ishibashi, Tsunenobu Shigematsu and Yasuharu Nakagawa*

(Ishibashi Laboratory)

Vanadium in sea-water has been studied by Ernst and Hörman (Nathr, Ges. Wiss. Göttingen. (1936) (ii) 1). They determined V spectroscopically with the material concentrated with  $\text{Fe}(\text{OH})_3$  as carrier, and found 0.3  $\gamma$  of V per L sea-water.

The authors quantified V in sea-water colorimetrically by phosphotungstate me-

thod as follows: 5~10 L of sea-water was taken, acidified with 20 ml of HCl and added 20 mg of  $\text{Fe}^{+++}$  as  $\text{FeCl}_3$  solution. Then  $\text{Fe}^{+++}$  was precipitated with cupferon solution (5 g of cupferon), allowed to stand for 2~3 days. Then V was coprecipitated with Fe-cupferon nearly completely. The precipitate was filtered, ashed in platinum crucible and fused with small amounts of  $\text{Na}_2\text{CO}_3 + \text{K}_2\text{CO}_3$ . The fused mass was extracted with hot water. The solution was acidified with  $\text{HNO}_3$ , added a drop of  $\text{Br}_2$  water, and evaporated to dryness. The dried material was extracted with water, and the solution transferred to 10 ml measuring flask. Then added 1 ml of  $\text{HNO}_3(1:1)$ ,  $\text{H}_3\text{PO}_4$  (85%, 1:4) and  $\text{Na}_2\text{WO}_4$  solution (0.2 M) respectively. Diluted to the mark and the resultant color of the solution measured with Purfrich-photometer by using filter  $\text{S}_{43}$ . In this V determination,  $\times 40$  Co,  $\times 40$  Ni,  $\times 50$  Cu,  $\times 20$  U,  $\times 10$  Mo and  $< 3$  mg Fe did not interfere.

Vanadium in the sea-water off shore of Shirahama, Wakayama Prefect., Japan was found to be 3~4  $\gamma/\text{L}$ , and the mean value 3  $\gamma/\text{L}$ .

The authors are indebted to Dr. T. Tokioka, for the help of sampling of sea-water.

## 9. Analysis of the Mixture of Thiourea and Ammonium Thiocyanate

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and Toshiaki Toba*

(Kodama Laboratory)

On the analysis of the mixture of  $\text{CS}(\text{NH}_2)_2$  and  $\text{NH}_4\text{SCN}$  studies have been made by Volhard, Reynolds & Werner, Storch, Krall, Gilfillan and Burrows<sup>1)</sup>. As their methods determining  $\text{CS}(\text{NH}_2)_2$  by iodometry and  $\text{NH}_4\text{SCN}$  by titration with  $\text{AgNO}_3$  solution were found to be unsatisfactory, following method is proposed, which determines  $\text{CS}(\text{NH}_2)_2$  by precipitation with xanthidrol-methanol solution and  $\text{NH}_4\text{SCN}$  by titration of the filtrate with  $\text{AgNO}_3$  solution.

1. Analysis of a sample which contains only thiourea. The methanol solution of a sample is taken into a 500 cc. beaker, and a mixture of 2 % xanthidrol-methanol solution and glacial acetic acid, in which the former solution contains xanthidrol in the ratio 2~3 mol per 1 mol  $\text{CS}(\text{NH}_2)_2$  in the sample solution and the quantity of the latter is 4 times in volume as much as that of the former solution, is added to the sample solution and agitated with a mechanical stirrer for 3 hrs. The beaker is then allowed to stand over night and the content is filtered through a glass filter. The precipitate is washed several times with methanol and water alternately, and in that case each of methanol and water is consumed 30 times in volume as much as the sample solution. The precipitate is dried for 1 hrs. at 110°C and is weighed